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2009/08/26 :
CIA-RDP88-00904R000100110

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2009/08/26 :
CIA-RDP88-00904R000100110



**Third United Nations
International Conference
on the Peaceful Uses
of Atomic Energy**

A/CONF.28/P/353
USSR
May 1964
Original: RUSSIAN

Confidential until official release during Conference

HETEROGENOUS OXIDATION OF UO_2 AND URANIUM
LEACHING PROCESSES IN ACID SOLUTIONS

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The processes of uranium leaching from ores are very complicated. However the main laws of these processes, as it is shown in the work, are connected with the kinetics of oxidation and solution of uranium oxides. Therefore the peculiarities of heterogenous oxidation processes and dissolving of UO_2 in acid solutions are of great interest.

The following questions were considered in the previously published works: the heterogenous processes of interaction of UO_2 and MnO_2 ¹⁾, catalytic influence of ions of iron²⁾, influence of ion composition of $Fe_2(III)$ and concentration of ions of hydrogen upon the oxidation and solution of UO_3 ³⁾. The present work deals with the new experimental data and gives certain general conclusions.

I. Dissolving of UO_2 in Sulphuric Solution Using
Oxidizers

Now we shall consider the substances of those numerous oxidizers, which can be employed in dissolving UO_2 in sulphuric acids, that are of great importance to the uranium industry, that is nitric acid, chloric acid, manganese bioxide as well as ions of iron (III).

(a) Nitric Acid

In case when the nitric acid is employed in sulphuric solutions at low temperatures, its oxidizing properties become apparent only in relatively concentrated solutions of H_2SO_4 .

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The oxidizing properties of this substance increase if the concentration of the nitric acid becomes higher. (See Table 1).

Table 1

Degree of Dissolving of Uranium vs Concentration
of Sulphuric and Nitric Acids

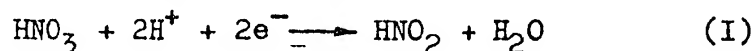
Concentration of H_2SO_4 , M	Degree of dissolving (%) at concentration of HNO_3 , M			
	-	0.032	0.094	0.186
0.05	1.0	1.0	1.0	1.0
0.10	1.0	1.1	1.1	1.0
0.26	1.0	1.1	1.0	1.8
0.51	0.9	1.1	1.0	3.1
1.02	0.9	1.1	1.9	7.2
2.04	1.0	18.0	27.0	38.0

The experiments were carried out in an autoclave, UO_2 batch - 5 g , temperature - $20^\circ C$, duration of the experiment - 3 hours, the nitric acid was introduced in stoichiometric quantity.

The experiments concerning the influence of the temperature were carried out in analogous conditions, but they lasted 1 hour. Figure 1 shows the experimental data. The oxidizing activity of the nitric acid as in case of increasing the concentration of HNO_3 and H_2SO_4 grows unevenly. The beginning of the intensive oxidizing action of the nitric acid in the given conditions corresponds to a temperature of $70^\circ C$, i.e. to a temperature when HNO_3 begins to decompose considerably⁴⁾.

These peculiarities of nitric acid are explained assuming that in definite conditions the process of autocatalytic decomposition of HNO_3 goes on rapidly, and the products of decomposition are strong oxidizers.

While examining the electro-chemical reduction of the nitric acid solutions of middle and high concentration Vetter ⁵⁾ came to a conclusion that the redox potential is in logarithmic dependence on concentration of the nitrous acid. On the contrary, Monk and Ellingham ⁶⁾ supposed that the reaction determining the potential is as follows:



i.e., the direct oxidizer - is the nitric acid. The existence of discrepant opinions necessitates further investigations of this question.

Figure 2 gives the results of simultaneous examination of the kinetics of dissolving of UO_2 and establishing of the potential in system $\text{UO}_2\text{-H}_2\text{SO}_4\text{-HNO}_3$ at 20°C .

When the nitric acid and uranium dioxide are introduced into the sulphuric solution the redox potential increases gradually and even after two hours cannot gain the constant value.

If we compare curves I and I^{I} , 2 and 2^{I} , 3 and 3^{I} , we shall see that there is no direct relation between the kinetics of establishing the redox potential and dissolving of the uranium dioxide. Meanwhile the oxidation and dissolution of UO_2 even in the solution containing 100 gr/l of HNO_3 reaches several per cent only after two hours, the value of redox potential increases up to 300mv.

These data can be explained if the nitric acid is not a direct oxidizer in the experimental conditions, and the products of its decomposition take part in the process of oxidation. That is these products that determine the value of redox potential of this system.

Kay and Stern⁴⁾ show that the decomposition rate of even 100% nitric acid is negligible when the temperature is below 70°C. Therefore we can assume that the dissolving of UO_2 is connected probably with the acceleration of the process of HNO_3 decomposition upon the catalytic influence of products of its reduction-nitric oxides.

As it may be seen from Figure 3 the processes of oxidation and dissolution of UO_2 in the same system at 70°C are considerably quicker than at 20°C. And the kinetics of establishing the redox potential considerably changes.

We consider only the qualitative characteristics of the kinetics of the process, as the heterogeneous system is exclusively complicated and consists of solid substance, solution and gas, the amount of the latter depending on a number of parameters. Besides it is possible to make very important conclusions concerning the properties of the nitric acid during its interaction with the uranium dioxide.

The examination of the factors determining the oxidizing activity of the nitric acid during its interaction with the uranium dioxide shows that HNO_3 is not a direct oxidizer. All the factors favourable for increasing the oxidizing activity of the nitric acid are simultaneously the factors accelerating its decomposition.

Therefore it is evident that when HNO_3 interacts with uranium oxides, one even several of its decomposition products simultaneously formed substances, for example HNO_2 or NO_2 are direct oxidizers.

(b) Chlorates

The process of oxidizing of uranium dioxide by chlorate at 20°C as well as homogenous oxidation of $\text{U(IV)}^{(7)}$ is very slow. Table II shows the results of the experiments when the concentration of H_2SO_4 is 100g/l.

The rate of the heterogenous process in the presence of the solution components Y and Z may be expressed according to the Mass Action Law by the equation:

$$W = kS[Y]^a [Z]^b \quad (2)$$

where S - the value of the solid phase surface,
a and b - the exponents corresponding to the order of the reaction for the given component. In case when one of the components is taken in considerable excess to the solid substance to be dissolved, then

$$W = k'S[Y]^a \quad (3)$$

where $k' = k(Z)^b \approx \text{const.}$

Let us assume that laws of kinetics of the homogenous oxidation of U(IV) in the sulphuric acid with the use of the chlorate may be spread to the corresponding heterogenous process of oxidation and identify $[Y]$ with the chlorate concentration and $[Z]$ with the concentration of the hydrogen ions. The homogenous reaction rate is expressed by the equation

$$W = k(\text{HClO}_3)^{1/3} \quad (4)$$

where HClO_3 - the concentration of non-dissociated chloric acid.

From the equations (3) and (4) we can find

$$W = k'S/Y^{1/3} = k'S/Y_0^{1/3} (1-\xi)^{1/3} \quad (5)$$

where ξ - the degree of interaction in moment τ .

According to Dolivo-Dobrovolsky⁸⁾ for the solution of monodispersed substances

$$S = S_0(1-\xi)^{2/3} \quad (6)$$

Therefore

$$W = k'S/Y_0^{1/3}(1-\xi) = W_0(1-\xi) \quad (7)$$

Taking into account that the reaction rate is low, in the first approximation

$$W = \frac{dx}{d\tau} = W_0(1-\xi) \approx W_0 \quad (8)$$

Therefore $\xi = x/x_0$ and it is determined by the equation

$$\xi = W_0\tau + \text{const.} \quad (9)$$

The value of const. depends on the content of U(VI) in the uranium dioxide. The experimental data prove the equation (9).

Table II

Kinetics of Dissolving of UO_2 at 20°C when the Stoichiometric Quantities of Chlorate are introduced.

Duration, hours	Degree of dissolving (4)	
	Experiment	Equation(9)
1.0	0.020	0.020
2.0	0.023	0.023
3.0	0.026	0.026
4.0	0.030	0.029
5.0	0.031	0.032
6.0	0.034	0.035

Table III shows the degree of dissolving of UO_2 in H_2SO_4 with the concentration 100 g /l at 80°C and in a stoichio-metric quantity of the chlorate. In this case the equation(8)

will be
$$\frac{d(\frac{x}{x_0})}{d\tau} = \frac{d\xi}{d\tau} = W'_0 (1 - \xi)$$

or

$$-\frac{d(1-\xi)}{d\tau} = W'_0 (1 - \xi)$$

and it follows that

$$-\lg \frac{(1-\xi)}{\tau} = W'_0 = \text{const} \quad (10)$$

Table III gives this relation together with the experi-mental data. The mean value $w'_0 = w'_0 \cdot 2.303 = 0.345$ (accor-ding to Table III),from where it follows that

$$\xi = 1 - 10^{-0.345\tau} \quad (11)$$

The values ξ determined by the equation (11) agree with the experimental data.

Table III

Checking of the Equations (10) and (11) by
Experimental Data (at 80°C)

τ , hours	ξ	$-\lg \frac{(1-\xi)}{\tau}$	$\xi_{\text{calculated}}$
0.5	0.332	0.35	0.328
1.0	0.590	0.39	0.548
2.0	0.790	0.34	0.795
3.0	0.875	0.30	0.908
4.0	0.960	0.35	0.958

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(b) Manganese Dioxide

In previously published work¹⁾ it was shown that with great excess of MnO_2 the reaction rate did not change in time but depended on the ratio of UO_2 to MnO_2 . With the stoichiometric quantity of MnO_2 the reaction is slowed down (Table IV) the influence of the temperature is negligible.

Table IV

Degree of Dissolving of UO_2 vs Duration of
Process and Temperature at its Interaction with MnO_2

$T, \text{min.}$	Value of ξ (%) at the temperature of ($^{\circ}\text{C}$)		
	20°	30°	40°
5	12.0	12.4	14.6
15	15.8	17.5	17.9
30	20.2	20.3	24.4
45	23.0	22.5	25.8
60	25.8	25.6	30.4
120	29.6	29.6	35.2
180	30.8	30.7	38.2
240	35.3	35.4	41.4
300	39.1	41.0	46.6
360	44.3	46.5	48.4

The comparison of the results of the sedimentometric analysis of the polydispersed manganese dioxide shows the existence of conglomeration of particles in the sulphuric acid. The same conglomerates are formed in the complex containing UO_2 and MnO_2 . Probably under these conditions

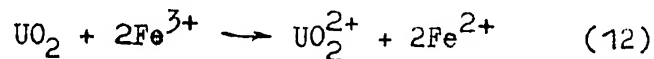
the process rate is not determined by the chemical interaction rate; the process takes place in the surface hydrated layers of UO_2 and MnO_2 particles which are in close contact with each other. As this contact depends upon the ratio of number of UO_2 and MnO_2 particles in conglomerates and their relative sizes, various kinetic regularities may be seen when MnO_2 is in stoichiometric quantity or in excess.

The interaction of UO_2 and MnO_2 in sulphuric solution proceeds relatively slow due to the decisive role of the particles contact, in this case the manganese dioxide is not in great excess.

(d) Ions of Trivalent Iron

An important role of ions of Fe(III) in the processes of leaching of uranium ores is considered in some works^(2,9-12). But certain regularities of interaction of UO_2 and Fe(III) are not investigated sufficiently.

Recently published works⁽³⁾ describe the relation between the interaction degree and value of pH at a definite duration of the process. Usually it is assumed that this process takes place in accordance with the equation



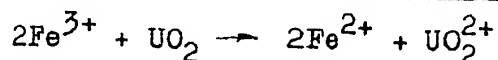
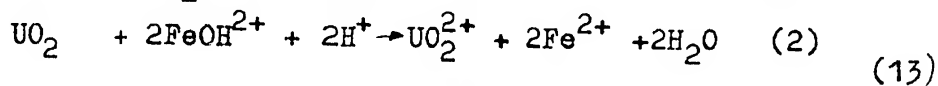
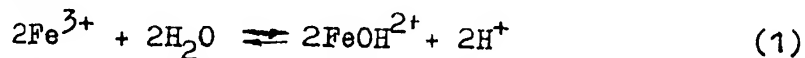
but if only non-hydrolyzed ion of Fe^{3+} takes part in it, then the influence of pH is not marked.

As it was shown in the work by Betts, the rate of U(IV) oxidation with the help of iron(III) in the chloric acid decreases when the acid concentration increases from 0.105 to 1.021 M. The analogous phenomenon was noticed ¹⁴⁾ earlier, when neptunium(IV) was oxidized by Fe(III); and the concentration of hydrogen ions increased from 0.5 to 1.0 M.

The authors explained this influence of the concentration of the hydrogen ions by the fact that hydrolyzed ions of Fe(III) namely FeOH^{2+} and $\text{Fe}(\text{OH})_2^+$ took part in the process.

Evidently Fe(III) as an oxidizer in sulphuric solutions keeps the same properties as in the case of the heterogeneous processes.

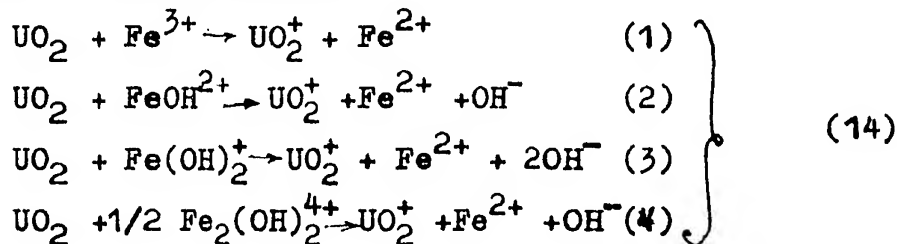
When assuming this the process expressed by the summary equation (12) consists of two consequent reactions, as it is shown in the scheme for oxidation by FeOH^{2+} given below,



Special experiment aimed at the investigation of the influence of the solvent flow rate on the interaction of UO_2 (fixed pressed tablets) with the sulphuric solution, containing Fe(III) showed that in definite conditions the process was in the kinetic area. This conclusion may be applied for the further experimental data.

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The reversible reaction(13.1) proceeds rapidly, therefore, probably the rate of the total process is determined by one of the stages of the heterogenous oxidation(13.2). It means that the rate of uranium dioxide dissolution must be propotional to the concentration of ions of FeOH^{2+} or other ions of Fe(III) . In other words, the kinetics of interaction of UO_2 with ions of Fe(III) is determined by the rate of the processes:



if the reaction under consideration is of the first order in relation of Fe(III) .

When this assumption takes place, the rate of reaction (14.1)-(14.4) will be expressed by the equations:

$$\left. \begin{aligned} W_1 &= k_1 S / \text{Fe}^{3+} & (1) \\ W_2 &= k_2 S / \text{FeOH}^{2+} & (2) \\ W_3 &= k_3 S / \text{Fe(OH)}_2^+ & (3) \\ W_4 &= k_4 S / \text{Fe}_2(\text{OH})_2^{4+}^{1/2} & (4) \end{aligned} \right\} \quad (15)$$

If we express the concentrations of all the ions of Fe(III) as $/ \text{Fe}^{3+} /$ using the equation (15) and taking into account

$$W = W_1 + W_2 + W_3 + W_4 \quad (16)$$

then

$$W = \left(k_1 + \frac{k_2 K_1}{/ \text{H}^+ /} + \frac{k_3 K_1 K_2}{/ \text{H}^+ /^2} + \frac{k_4 K_d^{1/2} K_1}{/ \text{H}^+ /} \right) S / \text{Fe}^{3+} / , \quad (17)$$

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where K_1 and K_2 - the first and the second constants of the hydrolysis of Fe^{3+} , respectively, and K_d - the constant of the dimerization of FeOH^{2+} . If the concentration of hydrogen ions is constant, the equation (17) will be:

$$W = k'S/\text{Fe}^{3+} \quad (18)$$

Assuming that the process under consideration is a reaction of the second order in relation to Fe(III) it is easy to become convinced that

$$W = \left(k_1 + \frac{k_2 K_1^2}{[\text{H}^+]^2} + \frac{k_3 K_1^2 K_2^2}{[\text{H}^+]^4} + \frac{k_4 K_d K_1^2}{[\text{H}^+]^2} \right) S/\text{Fe}^{3+}{}^2 \quad (19)$$

and if the concentration of hydrogen ions is constant, then

$$W = k''S/\text{Fe}^{3+}{}^2 \quad (20)$$

The ionic composition of Fe(III) solutions essentially depends on the concentration of hydrogen ions, therefore the equations (17) and (19) cannot be applied directly. It is possible, however³⁾, to show that in the area of pH where ferric hydroxide does not yet settle.

$$[\text{Fe}^{3+}] = -\frac{(\text{H}^+)}{4K_d K_1} \left(\frac{(\text{H}^+)}{K_1} + \frac{K_2}{[\text{H}^+]} + 1 \right) - \sqrt{\left(\frac{(\text{H}^+)}{K_1} + \frac{K_2}{[\text{H}^+]} + 1 + 8K_d \text{Fe(III)} \right)} \quad (21)$$

It follows from this equation that when the concentration of hydrogen ions increases, the value of $[\text{Fe}^{3+}]$ increases.

Due to it, as it is clear from the equations (17)-(20), the reaction rate at a definite value of pH should pass its maximum because the values of k' and k'' are decreased with the increase of hydrogen ions concentration.

It is difficult to integrate the equations (18) and (20) because not only $/\text{Fe}^{3+}/$ concentration but the value of S are the function of the process duration. The rate of any process of dissolution of a solid substance may be expressed with the equation (if the process is in kinetics area).

$$W = kS f(c) \quad (22)$$

where $f(c)$ is the function of the reacting substance concentration. Besides, the value of surface S is the function of the mass of the dissolved solid substance. Therefore

$$W = - \frac{dm}{d\tau} = k f(c) f(m), \quad (23)$$

or

$$- \frac{dm}{f(m)} = kf(c) d\tau$$

If we sign $c = \mathcal{C}(\tau)$ and $f(c) = f[\mathcal{C}(\tau)] = \mathcal{F}(\tau)$, (24)

then
$$- \frac{dm}{f(m)} = k \mathcal{F}(\tau) d\tau$$

or

$$k = \frac{\int_m^{m_0} \frac{dm}{f(m)}}{\int_0^\tau \mathcal{F}(\tau) d\tau} \quad (25)$$

For the floating bodies of ball form the equation

$$\int_m^{m_0} \frac{dm}{f(m)} = \frac{3\rho_s^{2/3}}{(36\pi)^{1/3}} (m_0^{1/3} - m^{1/3}), \quad (26)$$

and as $m = m_0 (1 - \xi)$, then

$$\int_m^{m_0} \frac{dm}{f(m)} = \frac{3\rho_s^{2/3} m_0^{1/3}}{(36\pi)^{1/3}} [1 - (1 - \xi)^{1/3}] = A [1 - (1 - \xi)^{1/3}] \quad (27)$$

The value $\int_0^\tau \mathcal{F}(\tau) d\tau = \int_0^\xi f(c) d\tau$ can be found graphically by integral at the right choice of the function $f(c)$ that is checked by the independence of value of k on the initial concentration.

The quantitative treatment of the experimental data for

the interaction of monodispersed UO_2 with Fe(III) can be done by the equation:¹⁾

$$k' = \frac{A[1-(1-\epsilon)^{1/3}]}{\int_0^\tau F(\tau) d\tau}, \quad (28)$$

$F(\tau) \equiv f(c) \equiv [\text{Fe}^{3+}]$ if the reaction is of the first order in relation to Fe(III) , and $F(\tau) = f(c) \equiv c^2$, if the reaction is of the second order. With $F(\tau) = [\text{Fe}^{3+}]$ the constant k' does not depend on the initial concentration of Fe(III) . By this method it was proved that the reaction is of the second order in relation to the ions of iron. It is evident that U(VI) is formed by disproportioning of U(V) . As it was shown in the work¹⁵⁾ the disproportioning of U(V) in sulphuric solutions is a rapid process.

As k_1 is a function of the concentration of hydrogen ions, then according to the equation (17) it is possible to evaluate the process rate constants (14.1)-(14.4). It is found that $k_1 \approx 1$, $k_2 \approx 10$, $k_3 \approx 10000$, and $k_4 \approx k_1$. These results prove the assumption that the hydrolyzed ions of Fe(III) are of great importance for the oxidation and dissolving of UO_2 .

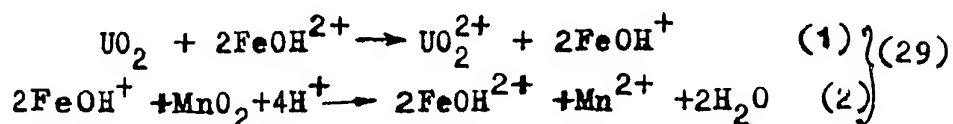
2. Regularities of UO_2 Dissolving in Sulphuric Solution When Various Oxidizers and Ions of Iron Take Part in the Process

As it was previously noted in real conditions of uranium leaching from the ores, the solutions practically always contain the ions of iron, which influence considerably on the process of oxidation and dissolving of primary uranium minerals. In this connection let us consider certain regularities of dissolving, when the pH value varies widely with the use of the nitric acid, manganese dioxide, calcium chlorate as oxidizers and adding Fe(II) .

* The deduction of the equation is omitted because of its laborious

The oxidizers were introduced in stoichiometric quantity in relation to the uranium dioxide, and the concentration of Fe(II) (0.5g/l) at the given amount of the solution corresponded to approximately 12 per cent of the necessary amount of Fe(III) for oxidizing UO_2 . The pH value was kept constant during the experiment. The experiments were carried out at $25^{\circ}C$ and lasted 1 hour³⁾.

Figure 6 shows the influence of Fe(III), MnO_2 , HNO_3 and $KClO_3$ as oxidizers on uranium recovery into the solution. The course of curves 1 and 2 given in Figure 6 is analogous because the dissolving of uranium is determined by the same process in both cases. The interaction of ions of Fe(III) and UO_2 is not advisable to express by the equation (12); more exactly, this scheme is suitable, when $pH < 1$ only. At the other pH value the hydrolyzed ions of Fe(III) are of the predominant importance and the reaction proceeds, for example, according to the scheme.

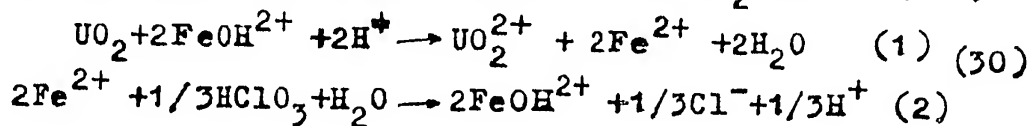


The reaction (29.2) proceeds rapidly and the rate of UO_2 dissolving and consequently at definite duration of the process the degree of UO_2 dissolving are determined by its interaction with hydrolyzed ions of Fe(III). In other words, the ions of iron in this process act as inductors as it is shown in the work by Shilov¹⁶⁾.

As we can see from Figure 6 the maximum of curves 1 and 2 does not coincide somewhat. They correspond

to pH equal to 1.5 and 1.8. Probably it is explained by different concentration of Fe(III) taking part in the process under consideration when Fe(III) is introduced in stoichiometric quantity.

Weakly noticed maximum on curve 3 is located at lower value pH if compared with curve 1. If the dissolving of UO_2 with using chlorate as an oxidizer and adding Fe(II) proceeded like the dissolving when MnO_2 and Fe(II) are used, the position of the maximum on the curve and the maximum value ϵ would be equal in both cases. The difference becomes clear at the assumption that the process rate (30.2) is lower than the rate of the interaction of UO_2 and Fe(III)



It is evident that the processes similar to (29.2) or (30.2) do not proceed in the conditions under consideration when HNO_3 and $HNO_3 + Fe(II)$ are used as oxidizers (curve 4).

These peculiarities of dissolving of UO_2 in sulphuric solutions with using oxidizers and ions of iron explain the regularities of leaching of uranium ores with using the same oxidizers.

As it is known, the attempts to use the nitric acid as oxidizer in leaching processes without heating failed¹⁷⁾. It is completely in accordance with the behaviour of nitric acid in the heterogenous process of oxidation and with dissolving of UO_2 in sulphuric solutions considered above. The use of the

nitric acid when the uranium is leached of ores with heating up to $80-90^{\circ}\text{C}$ is effective, the fact being explained by the autocatalytic decomposition of HNO_3 under the influence of the products of its reduction having high oxidizing activity. The chlorates are used in the acid leaching mainly without heating but the process is very prolonged. The oxidation and dissolving of UO_2 in the sulphuric solution with the use of this oxidizer and ions of iron proceed likewise.

Comparatively rapid dissolving of UO_2 with the use of MnO_2 and ions of iron explains the importance of using of this oxidizer in the uranium hydro-metallurgy.

Together with it, another factor—the rate of the diffusion along the capillaries, pores of ores—becomes of great importance during the leaching of uranium ores in the conditions of the effective oxidation of its primary minerals. In the cases, when the access of the reagent to the uranium minerals is free, the optimum area of pH (Figure 6) and the conditions of leaching of ores practically coincide. But in majority of cases, the leaching is carried out at lower pH value. This is explained by the fact that the lowering of the rate of uranium minerals dissolving at such values of pH is compensated possibly by the dissolving of the rock and the acceleration of the process connected with it.

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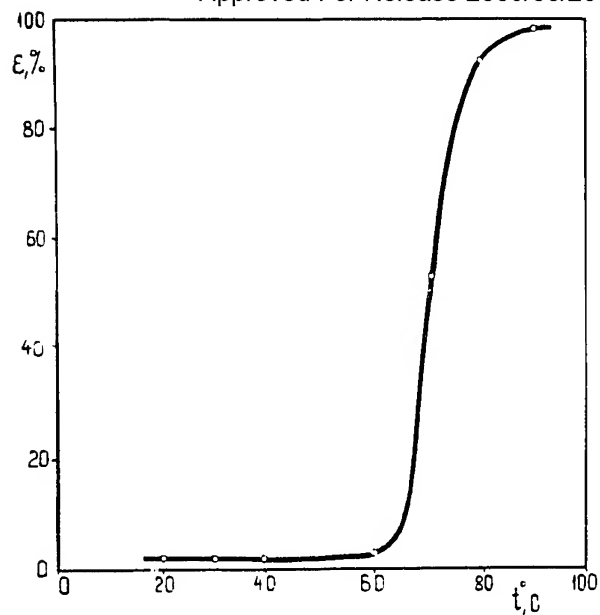


Fig. 1

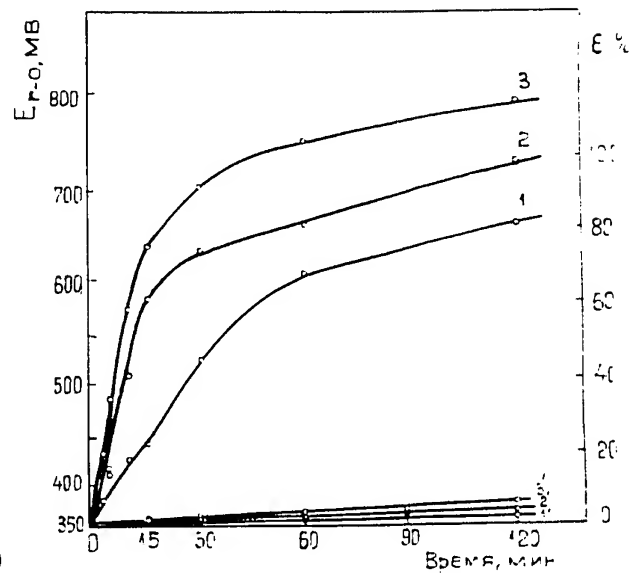


Fig. 2

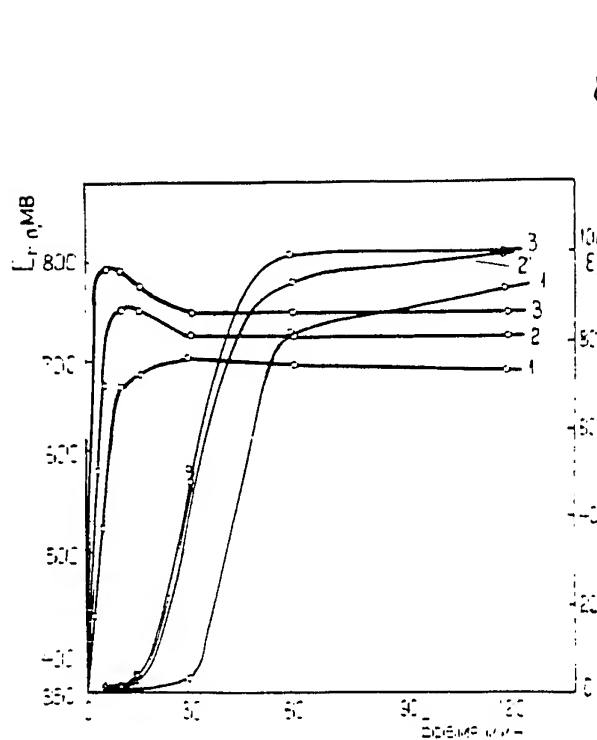


Fig. 3

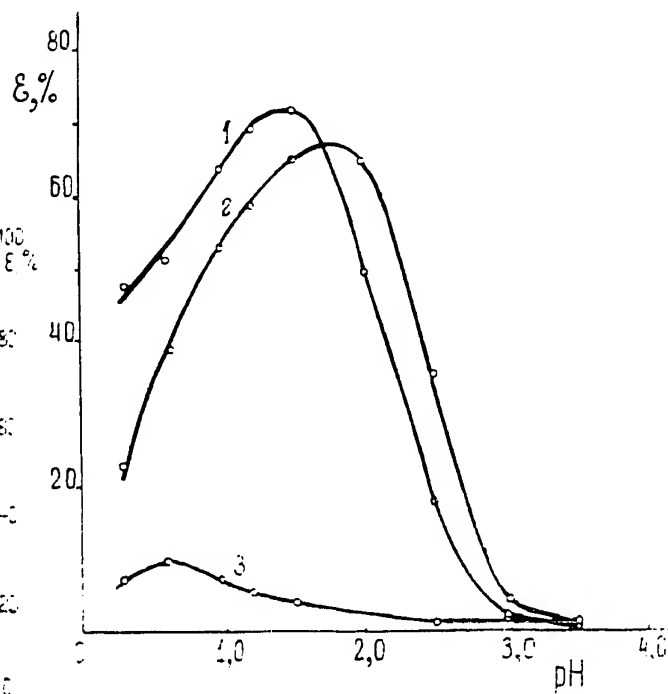


Fig. 4

C a p t i o n s

Figure 1. Degree of interaction of UO_2 and nitric acid in H_2SO_4 at a concentration of 10g/l vs temperature.

Figure 2. Value of the redox potential of system $\text{H}_2\text{SO}_4 - \text{HNO}_3 - \text{UO}_2$ (curves 1, 2, 3) and degree of uranium dissolving (curves 1', 2', 3') vs concentration of HNO_3 and duration of process at 20°C. Curves 1 and 1' correspond to $\text{HNO}_3 - 20\text{g/l}$, 2 and 2' - 60g/l , 3 and 3' - 100g/l , concentration of $\text{H}_2\text{SO}_4 - 45\text{g/l}$.

Figure 3. Value of the redox potential of system $\text{H}_2\text{SO}_4 - \text{HNO}_3 - \text{UO}_2$ (curves 1, 2, 3) and degree of uranium dissolving (curves 1', 2', 3') vs concentration of HNO_3 and duration of process at 70°C. Curves 1 and 1' correspond to $\text{HNO}_3 - 10\text{g/l}$, 2 and 2' - 25g/l , 3 and 3' - 60g/l , concentration of $\text{H}_2\text{SO}_4 - 45\text{g/l}$.

Figure 4. Degree of UO_2 dissolving vs value of pH with using oxidizers and adding of Fe(II) :

1 - Fe(III) ;	3 - $\text{KClO}_3 + \text{Fe(II)}$;
2 - $\text{MnO}_2 + \text{Fe(II)}$;	4 - $\text{HNO}_3 + \text{Fe(II)}$.